PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2003-288737
(43)Date of publication of application : 10.10.2003

(51)Int.Cl. G11B 7/24
B41M 5/26

(21)Application number : 2002-130158
(22)Date of filing : 01.05.2002

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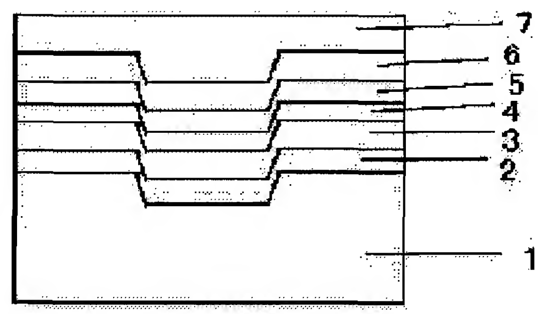
(30)Priority
Priority number : 2002017389 Priority date : 25.01.2002 Priority country : JP

(54) OPTICAL DATA RECORDING MEDIUM

(57)Abstract:
PROBLEM TO BE SOLVED: To realize an initialization-free medium, having a recording density of 4.7 GB/disc-120 mm-in-diameter or larger, which responds to high-speed overwrite recording, and is compatible with DVD-ROMs.
SOLUTION: An optical recording medium, having a recording layer including Sb and Te, in addition, substantially no other elements or at least one of elements of group 1 to group 7 in the periodic table and remnant layers is a phase-change recording medium, wherein the remnant layers include a material for speeding up linear velocity, and by a recording operation through energy irradiation to the optical data medium, the other elements in an amount existed in the recording layer at end of a deposition process of the recording layer or more are migrated from the remnant layers into the recording layer and exist there.



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CLAIMS

[Claim(s)]
[Claim 1]When a layer of said the complementary carries out an energy ray to said optical information medium and carries out recording operation to it in an optical recording medium characterized by comprising the following including high linear velocity-ized material, A phase change recording medium with which said other elements of quantity of the more than which existed in this recording layer at the time of an end of a film production process of this recording layer shift into a recording layer, and come to exist from a layer of said the complementary.
other elements other than Sb and Te are not included substantially -- the [or / the Ith fellows of the periodic table thru/or] -- a recording layer containing at least one kind of element of an element belonging to VII fellows.
A layer of the complementary.

[Claim 2]The phase change recording medium according to claim 1 with which said recording layer is characterized by Sb and being germanium content of 0 thru/or less than pentatomic % in an atomic number, including 1/3 or less Te of Sb.
[Claim 3]The phase change recording medium according to claim 1 or 2, wherein a layer of said the complementary is a crystallization promoting layer containing a recorded state stabilizing material and a crystallization accelerating material.
[Claim 4]The phase change recording medium according to claim 3, wherein said recorded state stabilizing materials are four group elements, 1B group element, three group elements, and/or five group elements.
[Claim 5]The phase change recording medium according to claim 4, wherein said recorded state stabilizing materials are germanium, Cu, In, B, and/or N.
[Claim 6]The phase change recording medium according to claim 3, wherein said high linear velocity-ized materials are Ga, Dy, Ca, Mg, and at least one element chosen from a group which consists of Mn.
[Claim 7]The phase change recording medium according to claim 6, wherein said high linear velocity-ized material is Ga or/and Mn.
[Claim 8]The phase change recording medium according to claim 3, wherein said crystallization accelerating materials are five group elements and six group elements.
[Claim 9]The phase change recording medium according to claim 8, wherein said crystallization accelerating materials are Sb, Bi, and/or Te.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]The initialization operation of this invention is unnecessary, it is excellent in preservation reliability, and relates to the optical information recording medium which can respond to high-speed over-writing record.
It is applied to an optical disc.

[0002]
[Description of the Prior Art]By laser beam exposure, by record of information, reproduction, and an eliminable optical information recording medium and a laser beam exposure as an optical information recording medium in which record of information, reproduction, and elimination are possible, The commercialization as phase change CD-RW using the reversible phase change of a crystallized state and an amorphous (amorphous) state, DVD-RAM, -RW, +RW media, etc. is expected.
[0003]This invention persons have furthered research and development about what was constituted from a recording layer having a metastable Sb3Te phase which belongs a recording layer to space group Fm3m among the above-mentioned phase change type optical information recording media (statements, such as JP,10-217069,A). This metastable phase differs from the recording layer of Sb-Te eutectic crystal structure, it does not separate into Sb and Sb2Te3, and disorder of the recording mark resulting from the grain boundary is not produced, either. Therefore, the thing using the recording layer having a metastable Sb3Te phase belonging to space group Fm3m has the strong point whose high density recording becomes possible. Repeatedly, since it is strong to the thermal shock at the time of record, the recording layer having a metastable Sb3Te phase belonging to space group Fm3m has the outstanding repetition recording characteristic.
[0004]By the way, in the phase change type optical information recording medium using the recording layer having a metastable Sb3Te phase belonging to this space group Fm3m, a recording layer is produced by the vacuum producing-film methods, such as sputtering and vacuum evaporation, and the film immediately after film production has usually become an amorphous state. On the other hand, the recording layer of the optical information recording medium produced commercially must be a crystallized state. In the erasable light information record carrier using a phase transition, it is because record film is set as an amorphous state by a recorded state and is generally set as a crystallized state in the state of elimination (initialization). For this reason, the initialization process which heat-treats a laser beam exposure etc. and crystallizes a recording layer immediately after film production of a recording layer was required. However, since the time for 30 seconds or more is required for the above-mentioned initialization process, a throughput is downed, it is [many devices for an initialization process / stand] necessary for [when mass-producing], and an installation cost becomes expensive. As a result, the rise of product cost will be caused.
[0005]The recording layer which contains at least one kind of element of the element which belongs to the Ith fellows of the periodic table thru/or VII fellows other than Sb and Te excluding other elements substantially to such a problem, In the optical recording medium which has a layer of the complementary which consists of a crystallization promoting layer which consists of a crystallization accelerating material and a recorded state stabilizing material, Said other elements of the quantity of the more than which existed in this recording layer at the time of the end of stage film formation of this recording layer by carrying out an energy ray to said optical recording medium, and carrying out recording operation to it shift into a recording layer, and it is made to exist from the layer of said the complementary, There is art which made the initializing step unnecessary (the application-for-patent No. 319887 [2001 to] specification). However, while the use to high-density image recording expands a phase change recording medium in recent years, much more high-speed over-writing realization (7 which is 2-5X of the reproduction linear velocity of DVD-ROM - 17 m/s) has come to be required. Although the improvement (following, improvement in the speed) in the crystallization rate of the recording layer material in the time of mark elimination (the melting point near [i.e.,]) is needed for high-speed over-writing realization here, it is difficult to deal with such high-speed over-writing in said art. Because, according to the knowledge known until now, can attain improvement in the speed of recording layer material by making the compounding ratio of Sb high about the phase change type optical information recording medium (JP,2000-43415,A) which has a metastable Sb3Te phase to which a recording layer belongs to space group Fm3m, but. Since the preservation reliability of a medium falls in connection with it, it is a practical level and is because the medium [over-write / medium / high-speed] cannot be provided.
[0006]
[Problem(s) to be Solved by the Invention]Therefore, the purpose of this invention cancels the above-mentioned problem, and there is in realizing the initialization loess media of the storage density of 120 or more mm disks of 4.7 GB / diameters in which the compatibility with DVD-ROM which can respond to high-speed over-writing record is possible.
[0007]
[Means for Solving the Problem]an aforementioned problem does not contain substantially other elements other than (1) "Sb of this invention, and Te -- the [or / the Ith fellows of the periodic table thru/or] -- in an optical recording medium which has a recording layer containing at least one kind of element of an element belonging to VII fellows, and a layer of the complementary, When a layer of said the complementary carries out an energy ray to said optical information medium and carries out recording operation to it including high linear velocity-ized material, "A phase change recording medium with which said other elements of quantity of the more than which existed in this recording layer at the time of an end of a film production process of this recording layer shift into a recording layer, and come to exist from a layer of said the complementary", and (2) Said recording layer Sb, A phase change recording medium given in said ** (1) paragraph characterized by being germanium content of 0 thru/or less than pentatomic % by an atomic number including 1/3 or less Te of Sb", (-- 3) "phase change recording medium given in said ** (1) paragraph or a ** (2) paragraph, wherein a layer of said the complementary is a crystallization promoting layer containing a recorded state stabilizing material and a crystallization accelerating material". (-- 4) "phase change recording medium given in said ** (3) paragraph, wherein said recorded state stabilizing materials are four group elements, 1B group element, three group elements, and/or five group elements", and (5) -- "-- said recorded state stabilizing material -- germanium, [Cu, In and] A phase change recording medium given in said ** (4) paragraph being B and/or N", (-- 6) "phase change recording medium given in said ** (3) paragraph, wherein said high linear velocity-ized materials are Ga, Dy, Ca, Mg, and at least one element chosen from a group which consists of Mn". (-- 7) "phase change recording medium given in said ** (6) paragraph, wherein said high linear velocity-ized material is Ga or/and Mn". (it is attained by 8) "phase change recording medium given in said ** (3) paragraph, wherein said crystallization accelerating materials are five group elements and six group elements", and (9) "a phase change recording medium given in said ** (8) paragraph, wherein said crystallization accelerating materials are Sb, Bi, and/or Te."
[0008]In an optical recording medium which has a recording layer containing at least one kind of element of an element which belongs to the Ith fellows of the periodic table thru/or VII fellows other than Sb and Te excluding other elements substantially, and a layer of the complementary,

Said other elements of quantity of the more than which existed in this recording layer at the time of an end of stage film formation of this recording layer by carrying out an energy ray to said optical recording medium, and carrying out recording operation to it shift into a recording layer, and it is made to exist from a layer of said the complementary, An aforementioned problem is solved by having made a layer of the complementary into a crystallization promoting layer containing a recorded state stabilizing material, a crystallization accelerating material, and high linear velocity-ized material.
[0009]
[Embodiment of the Invention]Hereafter, this invention is explained in detail. The feature of this invention is that the accessory constituent (this is also hereafter called "impurity") of the quantity of the more than which existed in the recording layer at the time of the end of stage film formation is a phase change recording medium which comes to exist in a recording layer by recording. It is a quantity contrastive with this accessory constituent, and means that Sb and Te in a recording layer maintain a Sb3Te system presentation, and the recording material of a recording layer holds the quantitative ratio of the grade in which phase-number conversion is possible to an amorphous phase-crystal phase by a write-operation and erasing operation as Sb as the main ingredients in this invention, and Te. The impurity of the quantity of the more than which said impurity was spread [in the crystallization promoting layer] to the recording layer including the recorded state stabilizing material at the time of phase change record as an impurity, and existed in the recording layer at the time of the end of stage film formation comes to exist in a recording layer. Therefore, the recorded state stabilizing material which a recording material should include beforehand can be decreased.
[0010]In this invention, high linear velocity-ized material is included in a crystallization promoting layer as an impurity. High linear velocity-ized material has an effect which raises the crystallization rate of a recording material [/ near the melting point], and the high-speed over-writing realization (7 which is 2-5X of the reproduction linear velocity of DVD-ROM - 17 m/s) of it is attained. Since improvement in the speed of recording layer material can be attained without increasing the rate of Sb occupied to a recording layer by adding high linear velocity-ized material, the problem that preservation reliability will fall with improvement in the speed of recording layer material does not arise.
[0011]It is preferred to use the group Ga, Dy, Ca, Mg, and at least one element chosen from Mn as a high linear velocity-ized material. The effect of improvement in the speed of recording layer material is checked from the result in which these investigated the addition effect over the phase change type optical information recording medium which has a Sb3Te phase about many elements on the periodic table.
[0012]a group -- Ga and/or especially Mn are especially preferred. Although each of D(ies), Ca, and Mg can attain improvement in the speed, when it is made to contain so much, it has the problem of causing the fall of a repetition recording characteristic. Although Ga has the problem of causing the fall of a recording characteristic repeatedly like Dy, Ca, and Mg, it has the effect of raising a conservation characteristic. For this reason, stabilization of a recorded state can also be attained with improvement in the speed. On the other hand, although the effect that Mn raises a conservation characteristic is not like Ga, the fall of a recording characteristic is not caused [the case where it is made to contain so much]. By using Ga and/or Mn as a high linear velocity-ized material from these things, a repetitive characteristic is good and it is possible to realize the phase change recording medium in which the conservation characteristic was further excellent.
[0013]When using germanium as a recorded state stabilizing material included in a crystallization promoting layer, germanium which a recording material is made to contain may be less than pentatomic %. When germanium which a recording material is made to contain in a crystal recording layer in a Sb3Te system recording layer is less than pentatomic %, crystallization temperature will be less than 160 **. When crystallization temperature is less than 160 **, formation of the crystal record film of 80% of relative reflectance is attained also in the membrane formation process below the plastic deformation temperature of a polycarbonate board by using a crystallization promoting layer. It is a recording material for crystal recording layer formation which germanium is less than three atom % more desirably, and does not contain germanium most desirably. Here, the case where germanium contained in a recording material is less than one atom % is defined as not containing germanium substantially. When germanium contained in a Sb3Te system recording material is 3atom%, the crystallization temperature of the case of 147 ** and less than one atom % is 110 **. In the case of 147 ** former crystallization temperature, at least 67 ** of the above-mentioned polycarbonate board heat-resistant temperature becomes usable crystal record film by initialization loess. The relative reflectance is about 80%. Becoming a crystal film even whose substrate temperature of 67 ** is still more perfect in the case of 110 ** latter crystallization temperature, the relative reflectance will be not less than 83%.
[0014]Record equivalent to at least 0.1 micrometer of record mark length is possible by extracting the beam system of recording laser light in this invention, since the recording material is a Sb3Te system recording material. As a recorded state stabilizing material in this invention, four group elements, 1B group element, and three group elements are effective. germanium is the most effective and also Cu, In, and B are effective. In addition, five fellows' N is also effective in recorded state stabilization. As a crystallization accelerating material, five group elements and six group elements are effective. In particular, Sb, Bi, and Te are effective.
[0015]A crystallization promoting layer contains one or more sorts of ******** of the above, a recorded state stabilizing material, a crystallization accelerating material, and high linear velocity-ized material. The layer which consists of 2 yuan, Bi and germanium, is the most preferred as a crystallization promoting layer. To a crystallization promoting layer, the further impurity element may be added for melting point adjustment etc. An usable material as an impurity element Ag, Cd, Ce, Co, Cr, Fe, H, Hg, Ir, K, La, Li, Mo, Na, nickel, O, P, Pb, Pd, Po, Pr, Pt, Pu, Rb, Rh, Ru, S, Se, Si, Sn, Sr, Th, Ti, Tl, U, Cl, Br, etc. are mentioned.
[0016]The above-mentioned crystallization promoting layer does not need to serve as a thin film [**** / completely] on a substrate. That is, in the case of about 1 nm, membrane formation thickness is the island shape of discontinuous a large number by mass membrane thickness. If membrane formation thickness increases, said islands will be connected and it will become a perfect thin film on a substrate. In this invention, said island shape is also called crystallization promoting layer in a microscopic meaning.
[0017]Next, an example of the optical information recording medium by this invention is shown in drawing 1. (1) is a substrate and (2) is the 1st dielectric layer and an organic protective layer by which, as for a reflection radiation layer and (7), the 2nd dielectric layer and (6) are provided [(3)] for a recording layer and (5) on a reflection radiation layer if needed in a crystallization promoting layer and (4). By a diagram, only the information board side of an optical information recording medium is shown. As for the material of a substrate, in almost all cases, polycarbonate is used. After substrate thickness forms the layer of drawing 1 in the case more than 1-mm thickness, and about 0.6-mm substrate, it is and, in more than 2.0-mm thickness, in the case of **0.3 mm and 1.0-mm thickness, the permissible error of the substrate thickness in this invention comes out of the case where paste other 0.6-mm boards together and it is referred to as about 1.2 mm, **0.2 mm in the case of **0.2 mm and 0.6-mm thickness. The slot is formed in the substrate and the depth is 200A - about 450A. The pitch of a slot is 0.74 micrometer when using it as DVD compatible media. If minuteness making of this pitch is carried out to about 0.3 micrometer, record of not less than 20 GB is possible on a disk 120 mm in radius by using the laser of blue light.
[0018]In this invention, as the 1st and 2nd dielectric layers, SiOx, ZnO, SnO_2, aluminum$_2O_3$, TiO_2, Metallic oxides, such as In_2O_3, MgO, ZrO_2, and Ta_2O_5, Carbide, such as sulfides, such as nitrides, such as Si_3N_4, AlN, TiN, BN, and ZrN, ZnS, and TaS_4, SiC, TaC, B_4C, WC, TiC, and ZrC, is mentioned. Such materials can be alone used as a protective layer, and can also be used as a mixture. For example, ZnS, SiOx, and Ta_2O_5 and SiOx are mentioned as a mixture.
[0019]The thickness of the 1st dielectric layer has the preferred range of 50-250 nm. If it becomes thinner than 50 nm, it becomes the fall of an environment-resistant protection feature, a heat-resistant fall, and the fall of ********, and is not desirable. in the film production process by a sputtering technique etc. if it becomes thicker than 250 nm -- film temperature -- since film peeling and a crack arise by the rise of a degree or the fall of the sensitivity at the time of record is brought about, it is not desirable. As for the thickness of the 2nd dielectric layer, 15-50 nm is preferred. In the case of the 2nd dielectric layer, if thinner than 10 nm, heat resistance falls and it is not desirable. On the contrary, if 100 nm is exceeded, repetitive overwriting characteristics will worsen due to the fall of the film peeling by the fall of recording sensitivity, and a rise in heat, modification, and heat dissipation nature.
[0020]As a reflection radiation layer (6), the simple substance or alloy of material mainly concerned with metal, such as aluminum, Au, Cu, Ag, Cr,

Sr, Zn, In, Pd, Zr, Fe, Co, nickel, Si, germanium, Sb, Ta, W, Ti, and Pb, can be used. It is important for this layer to make heat radiate efficiently, and 50-160 nm of thickness is preferred. When thickness is too thick, radiation efficiency is too large, if sensitivity worsens and is too thin, sensitivity is good, but repetitive overwriting characteristics worsen. As the characteristic, thermal conductivity is high and it is required with a high-melting point that adhesion with the charge of a protective layer material should be good etc.

[0021]One in the case of pasting together to drawing 2 and using a substrate about 0.6 mm thick is shown. About dielectric materials, a crystallization accelerating material, a recording material, and the charge of a reflective radiating material, it is the same as that of the case of said drawing 1. In the case of drawing 2, transmissivity of the translucent reflection radiation layer of (13) and the 1st recording layer of (11) is made not less than 50%.

[0022]The case where it has a two-layer recording layer on a substrate about 1 mm thick is shown in drawing 3. About dielectric materials, a crystallization accelerating material, a recording material, and the charge of a reflective radiating material, it is the same as that of the case of said drawing 1. Since the transmissivity of the 1st recording layer is adjusted in the case of drawing 3, the 1st dielectric layer of (22) and the 2nd dielectric layer of (25) may be made into the multilayered constitution of a dissimilar material.

[0023]In order to carry out optical recording to the optical information recording medium of such this invention, when forming AMORUFASU equivalent to a reference clock length n cycle, laser pulse irradiation of a time (n-1) is performed at the time of n cycle. For example, when forming AMORUFASU of the length of reference clock 5 cycle shown in drawing 4, it irradiates with 4 times of laser pulses. in recording-linear-velocity 8.5 m/s -- the case (DVD.) of record with 63.7 MHz of reference clocks ** laser radiation start time delay in 2.4X record, for example, a figure, -- 19 ns and ** leading pulse width -- for 6 ns, for 7 ns, 15 mW and ** erase power are performed at 8 mW, and, as for ** multi-pulse width, ** cooling pulse width performs ** cooling power for ** record power at 0.1 mW for 14.5 ns.

[0024]For example, the above-mentioned material and the optical information recording medium by composition are the semiconductor lasers whose wavelength is 635 or 650 nm, and record reproduction can be carried out using the pickup of NA0.6. As a record method, a modulation code can use EFM or an EFM+ [8 / 16RLL (2, 10)] method by Pulse Width Modulation, for example. In this case, a pulse is divided into the multi-pulse part of a leading pulse and after that. A multi-pulse part is for repeating heating and cooling and performing them. In this case, the relation of each power serves as heating (record) power > erase power > cooling power, and cooling power is lowered to a read-out power grade. Usually, linear velocity is performed by 3.5 - 8.5 m/s, and read-out power is performed at 1 mW or less.

[0025]

[Example]Hereafter, this invention is explained based on an example. The thin film of the following (1) - (5) is formed by sputtering process on 0.6-mm thickness and a polycarbonate board (the following, PC board) 5 inches in diameter.

(1) Dielectric layer:ZnS-SiO2 which is the 1st (being a mol ratio of a target 79.5:20.5); 220 nm of thickness.

(2) Crystallization promoting layer : the target which consists of a recorded state stabilizing material, a crystallization accelerating material, and high linear velocity-ized material was used. A presentation and thickness are shown in Table 1.

(3) Recording layer : **** for alloy targets of Sb3Te system material. A presentation is shown in Table 1. 16 nm of thickness.

(4) Dielectric layer:$ZnS-SiO_2$ which is the 2nd (being a mol% ratio of a target 79.5:20.5); 10 nm of thickness.

(5) reflection radiation layer: -- AlTi; -- 140 nm of thickness.

Substrate temperature at the time of recording layer membrane formation was performed at the temperature of the limit in which a polycarbonate board does not carry out plastic deformation. In this case, IR lamp output is controlled so that the thermo couple contacted to the substrate will be 100 **, and after monitor temperature with a thermoelectrical object is stabilized, the substrate is conveyed in the recording layer membrane formation room. It was 67 ** when the substrate temperature in the recording layer membrane formation room was measured. After the end of membrane formation, after carrying out the spin coat of the UV curing resin, curing formation was carried out by UV light exposure. Thus, the manufactured substrate was pasted together to other 0.6-mm boards in thickness, and was made into about 1.2-mm thickness.

[0026]The wavelength of 660 nm and the pickup head of NA0.65 were used, and an equivalent for the storage density set to 4.7 GB on diameter 120 mm disk with recording linear velocity 8.5 and the speed of 14 m/s was recorded. Also when writing one mark, it recorded with the multipulse system which repeats laser radiation and cooling. Although the amorphous mark of the length of the multiple of clock 1 cycle was formed, laser radiation of the same number of times as the pitch of the clock per mark length and cooling were performed. The ratio of the laser radiation power at the time of amorphous mark formation (record power) to the power at the time of crystal space formation (erase power) differs in an optimum value with the thickness and material of a crystallization promoting layer, and are (record power)/(erase power) =0.4 - 0.6.

[0027]A jitter is the value which **(ed) standard deviation of gap of the reading time of the boundary of a recording mark and a space by read clock 1 period time among a recording characteristic (unit %), and modulation is the value which **(ed) amplitude of 14T with the reflectance of 14T.

[0028](Examples 1-8, comparative examples 1-2) About Examples 1-8 and the comparative examples 1-2. The media reflectance immediately after manufacture and relative reflectance, recording linear velocity 8.5, the repetition record jitter properties evaluated by speed (an equivalent for 2.5 or 4X linear velocity DVD) of 14 m/s, and time for a record jitter to go up 1% at the time of 80 ** storage were evaluated. The result is shown in Table 2. Relative reflectance serves as a rule of thumb of the crystallization percentage of completion in a film production stage, and is defined by the following formulas here.

[0029]

[Equation 1]Relative reflectance =(media reflectance before record)/(reflectance of melting recrystallization formed at time of record) x100 (%)

[0030]And since according to the knowledge which the artificer acquired media reflectance is changed with record as this is less than 80%, the good media characteristic is not obtained.

[0031]As a result of evaluating, about Examples 1-8, the media reflectance could secure not less than 20%, and the relative reflectance could secure not less than 80%, and the endurance time of 100 hours could be secured as preservation reliability, and preservation reliability was good. The first time and the 1000 times record of recording linear velocity 8.5 and the repetition record jitter properties in the speed of 14 m/s are less than 10%.

The good characteristic was shown.

[0032]On the other hand, although preservation reliability was good, the repetition record jitter properties in recording-linear-velocity 8.5 m/s are less than 10% and the first time and 1000 times record showed the good characteristic in the comparative example 1, The first time and 1000 times record were over 15%, and the repetition record jitter properties in the recording linear velocity of 14 m/s were the very bad characteristic. Since there is no high linear velocity-ized material, this is considered because improvement in the speed of a recording material is insufficient.

[0033]In the comparative example 2, recording linear velocity 8.5 and the repetition record jitter properties in the speed of 14 m/s had dramatically bad preservation reliability, although the first time and 1000 times record are less than 10% and showed the good characteristic. Since the rate of Sb was increased for improvement in the speed of recording layer material and preservation reliability fell, this is considered.

[0034]

[Table 1]

	結晶化促進層材料	結晶化促進層膜厚/nm	記録層材料	混合後組成 / atom%				
				Bi	Ge	X	Sb	Te
比較例1	BiGe	2.0	Sb80Te20	4.4	6.6	0	71.2	17.8
比較例2	BiGe	2.0	Sb86Te14	4.4	6.6	0	76.5	12.5
実施例1	BiGeGa	2.0	Sb80Te20	3.7	3.7	Ga:3.7	71.2	17.8
実施例2	BiGeGa	2.0	Sb80Te20	4.4	3.3	Ga:3.3	71.2	17.8
実施例3	BiGeDy	2.0	Sb80Te20	3.7	3.7	Dy:3.7	71.2	17.8
実施例4	BiGeMg	2.0	Sb80Te20	3.7	3.7	Mg:3.7	71.2	17.8
実施例5	BiGeCa	2.0	Sb80Te20	3.7	3.7	Ca:3.7	71.2	17.8
実施例6	BiGeGa	1.5	Ge2Sb78Te20	4	4.3	Ga:2.2	71.2	18.3
実施例7	BiGeMn	2.0	Sb80Te20	3.7	3.7	Mn:3.7	71.2	17.8
実施例8	BiGeGaMn	2.0	Sb80Te20	3.3	3.3	Ga:2.2 Mn2.2	71.2	17.8

[0035]
[Table 2]

	製造直後のメディア反射率／相対反射率	80℃保管時に記録ジッターが1%上昇する時間	初回記録／ダイレクトオーバーライト1000回目のジッター（記録線速 8.5m/s）	初回記録／ダイレクトオーバーライト1000回目のジッター（記録線速 14m/s）
比較例1	20%／83%	100時間以上	10%未満／10%未満	15%以上／15%以上
比較例2	20%／82%	5時間未満	10%未満／10%未満	10%未満／10%未満
実施例1	20%／81%	100時間以上	10%未満／10%未満	10%未満／10%未満
実施例2	20%／85%	100時間以上	10%未満／10%未満	10%未満／10%未満
実施例3	20%／83%	100時間以上	10%未満／10%未満	10%未満／10%未満
実施例4	20%／83%	100時間以上	10%未満／10%未満	10%未満／10%未満
実施例5	20%／83%	100時間以上	10%未満／10%未満	10%未満／10%未満
実施例6	20%／81%	100時間以上	10%未満／10%未満	10%未満／10%未満
実施例7	20%／83%	100時間以上	10%未満／10%未満	10%未満／10%未満
実施例8	20%／81%	100時間以上	10%未満／10%未満	10%未満／10%未満

[0036]
[Effect of the Invention]As mentioned above, initialization loess manufacture of a Sb_3Te system recording material phase change archive medium is enabled by this invention so that clearly from detailed and concrete explanation, The extremely outstanding effect that the initialization loess media of the storage density of 120 or more mm disks of 4.7 GB / diameters in which compatibility with DVD-ROM is possible are realizable is done so.

[Translation done.]

TECHNICAL FIELD

[Field of the Invention]The initialization operation of this invention is unnecessary, it is excellent in preservation reliability, and relates to the optical information recording medium which can respond to high-speed over-writing record.
It is applied to an optical disc.

[Translation done.]

PRIOR ART

[Description of the Prior Art]By laser beam exposure, by record of information, reproduction, and an eliminable optical information recording medium and a laser beam exposure as an optical information recording medium in which record of information, reproduction, and elimination are possible, The commercialization as phase change CD-RW using the reversible phase change of a crystallized state and an amorphous (amorphous) state, DVD-RAM, -RW, +RW media, etc. is expected.
[0003]This invention persons have furthered research and development about what was constituted from a recording layer having a metastable Sb3Te phase which belongs a recording layer to space group Fm3m among the above-mentioned phase change type optical information recording media (statements, such as JP,10-217069,A). This metastable phase differs from the recording layer of Sb-Te eutectic crystal structure, it does not separate into Sb and Sb2Te3, and disorder of the recording mark resulting from the grain boundary is not produced, either. Therefore, the thing using the recording layer having a metastable Sb3Te phase belonging to space group Fm3m has the strong point whose high density recording becomes possible. Repeatedly, since it is strong to the thermal shock at the time of record, the recording layer having a metastable Sb3Te phase belonging to space group Fm3m has the outstanding repetition recording characteristic.
[0004]By the way, in the phase change type optical information recording medium using the recording layer having a metastable Sb3Te phase belonging to this space group Fm3m, a recording layer is produced by the vacuum producing-film methods, such as sputtering and vacuum evaporation, and the film immediately after film production has usually become an amorphous state. On the other hand, the recording layer of the optical information recording medium produced commercially must be a crystallized state. In the erasable light information record carrier using a phase transition, it is because record film is set as an amorphous state by a recorded state and is generally set as a crystallized state in the state of elimination (initialization). For this reason, the initialization process which heat-treats a laser beam exposure etc. and crystallizes a recording layer immediately after film production of a recording layer was required. However, since the time for 30 seconds or more is required for the above-mentioned initialization process, a throughput is downed, it is [many devices for an initialization process / stand] necessary for [when mass-producing], and an installation cost becomes expensive. As a result, the rise of product cost will be caused.
[0005]The recording layer which contains at least one kind of element of the element which belongs to the Ith fellows of the periodic table thru/or VII fellows other than Sb and Te excluding other elements substantially to such a problem, In the optical recording medium which has a layer of the complementary which consists of a crystallization promoting layer which consists of a crystallization accelerating material and a recorded state stabilizing material, Said other elements of the quantity of the more than which existed in this recording layer at the time of the end of stage film formation of this recording layer by carrying out an energy ray to said optical recording medium, and carrying out recording operation to it shift into a recording layer, and it is made to exist from the layer of said the complementary, There is art which made the initializing step unnecessary (the application-for-patent No. 319887 [2001 to] specification). However, while the use to high-density image recording expands a phase change recording medium in recent years, much more high-speed over-writing realization (7 which is 2-5X of the reproduction linear velocity of DVD-ROM - 17 m/s) has come to be required. Although the improvement (following, improvement in the speed) in the crystallization rate of the recording layer material in the time of mark elimination (the melting point near [i.e.,]) is needed for high-speed over-writing realization here, it is difficult to deal with such high-speed over-writing in said art. Because, according to the knowledge known until now, can attain improvement in the speed of recording layer material by making the compounding ratio of Sb high about the phase change type optical information recording medium (JP,2000-43415,A) which has a metastable Sb3Te phase to which a recording layer belongs to space group Fm3m, but. Since the preservation reliability of a medium falls in connection with it, it is a practical level and is because the medium [over-write / medium / high-speed] cannot be provided.

[Translation done.]

EFFECT OF THE INVENTION

[Effect of the Invention]As mentioned above, initialization loess manufacture of a Sb_3Te system recording material phase change archive medium is enabled by this invention so that clearly from detailed and concrete explanation. The extremely outstanding effect that the initialization loess media of the storage density of 120 or more mm disks of 4.7 GB / diameters in which compatibility with DVD-ROM is possible are realizable is done so.

[Translation done.]

TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention]Therefore, the purpose of this invention cancels the above-mentioned problem, and there is in realizing the initialization loess media of the storage density of 120 or more mm disks of 4.7 GB / diameters in which the compatibility with DVD-ROM which can respond to high-speed over-writing record is possible.

[Translation done.]

* NOTICES *

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1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

MEANS

[Means for Solving the Problem]an aforementioned problem does not contain substantially other elements other than (1) "Sb of this invention, and Te -- the [or / the Ith fellows of the periodic table thru/or] -- in an optical recording medium which has a recording layer containing at least one kind of element of an element belonging to VII fellows, and a layer of the complementary, When a layer of said the complementary carries out an energy ray to said optical information medium and carries out recording operation to it including high linear velocity-ized material, "A phase change recording medium with which said other elements of quantity of the more than which existed in this recording layer at the time of an end of a film production process of this recording layer shift into a recording layer, and come to exist from a layer of said the complementary", and (2) Said recording layer Sb, A phase change recording medium given in said ** (1) paragraph characterized by being germanium content of 0 thru/or less than pentatomic % by an atomic number including 1/3 or less Te of Sb", (-- 3) "phase change recording medium given in said ** (1) paragraph or a ** (2) paragraph, wherein a layer of said the complementary is a crystallization promoting layer containing a recorded state stabilizing material and a crystallization accelerating material". (-- 4) "phase change recording medium given in said ** (3) paragraph, wherein said recorded state stabilizing materials are four group elements, 1B group element, three group elements, and/or five group elements", and (5) -- "-- said recorded state stabilizing material -- germanium, [Cu, In and] A phase change recording medium given in said ** (4) paragraph being B and/or N", (-- 6) "phase change recording medium given in said ** (3) paragraph, wherein said high linear velocity-ized materials are Ga, Dy, Ca, Mg, and at least one element chosen from a group which consists of Mn". (-- 7) "phase change recording medium given in said ** (6) paragraph, wherein said high linear velocity-ized material is Ga or/and Mn". (it is attained by 8) "phase change recording medium given in said ** (3) paragraph, wherein said crystallization accelerating materials are five group elements and six group elements", and (9) "a phase change recording medium given in said ** (8) paragraph, wherein said crystallization accelerating materials are Sb, Bi, and/or Te."
[0008]In an optical recording medium which has a recording layer containing at least one kind of element of an element which belongs to the Ith fellows of the periodic table thru/or VII fellows other than Sb and Te excluding other elements substantially, and a layer of the complementary, Said other elements of quantity of the more than which existed in this recording layer at the time of an end of stage film formation of this recording layer by carrying out an energy ray to said optical recording medium, and carrying out recording operation to it shift into a recording layer, and it is made to exist from a layer of said the complementary, An aforementioned problem is solved by having made a layer of the complementary into a crystallization promoting layer containing a recorded state stabilizing material, a crystallization accelerating material, and high linear velocity-ized material.
[0009]
[Embodiment of the Invention]Hereafter, this invention is explained in detail. The feature of this invention is that the accessory constituent (this is also hereafter called "impurity") of the quantity of the more than which existed in the recording layer at the time of the end of stage film formation is a phase change recording medium which comes to exist in a recording layer by recording. It is a quantity contrastive with this accessory constituent, and means that Sb and Te in a recording layer maintain a Sb3Te system presentation, and the recording material of a recording layer holds the quantitative ratio of the grade in which phase-number conversion is possible to an amorphous phase-crystal phase by a write-operation and erasing operation as Sb as the main ingredients in this invention, and Te. The impurity of the quantity of the more than which said impurity was spread [in the crystallization promoting layer] to the recording layer including the recorded state stabilizing material at the time of phase change record as an impurity, and existed in the recording layer at the time of the end of stage film formation comes to exist in a recording layer. Therefore, the recorded state stabilizing material which a recording material should include beforehand can be decreased.
[0010]In this invention, high linear velocity-ized material is included in a crystallization promoting layer as an impurity. High linear velocity-ized material has an effect which raises the crystallization rate of a recording material [/ near the melting point], and the high-speed over-writing realization (7 which is 2-5X of the reproduction linear velocity of DVD-ROM - 17 m/s) of it is attained. Since improvement in the speed of recording layer material can be attained without increasing the rate of Sb occupied to a recording layer by adding high linear velocity-ized material, the problem that preservation reliability will fall with improvement in the speed of recording layer material does not arise.
[0011]It is preferred to use the group Ga, Dy, Ca, Mg, and at least one element chosen from Mn as a high linear velocity-ized material. The effect of improvement in the speed of recording layer material is checked from the result in which these investigated the addition effect over the phase change type optical information recording medium which has a Sb3Te phase about many elements on the periodic table.
[0012]a group -- Ga and/or especially Mn are especially preferred. Although each of D(ies), Ca, and Mg can attain improvement in the speed, when it is made to contain so much, it has the problem of causing the fall of a repetition recording characteristic. Although Ga has the problem of causing the fall of a recording characteristic repeatedly like Dy, Ca, and Mg, it has the effect of raising a conservation characteristic. For this reason, stabilization of a recorded state can also be attained with improvement in the speed. On the other hand, although the effect that Mn raises a conservation characteristic is not like Ga, the fall of a recording characteristic is not caused [the case where it is made to contain so much]. By using Ga and/or Mn as a high linear velocity-ized material from these things, a repetitive characteristic is good and it is possible to realize the phase change recording medium in which the conservation characteristic was further excellent.
[0013]When using germanium as a recorded state stabilizing material included in a crystallization promoting layer, germanium which a recording material is made to contain may be less than pentatomic %. When germanium which a recording material is made to contain in a crystal recording layer in a Sb3Te system recording layer is less than pentatomic %, crystallization temperature will be less than 160 **. When crystallization temperature is less than 160 **, formation of the crystal record film of 80% of relative reflectance is attained also in the membrane formation process below the plastic deformation temperature of a polycarbonate board by using a crystallization promoting layer. It is a recording material for crystal recording layer formation which germanium is less than three atom % more desirably, and does not contain germanium most desirably. Here, the case where germanium contained in a recording material is less than one atom % is defined as not containing germanium substantially. When germanium contained in a Sb3Te system recording material is 3atom%, the crystallization temperature of the case of 147 ** and less than one atom % is 110 **. In the case of 147 ** former crystallization temperature, at least 67 ** of the above-mentioned polycarbonate board heat-resistant temperature becomes usable crystal record film by initialization loess. The relative reflectance is about 80%. Becoming a crystal film even whose substrate temperature of 67 ** is still more perfect in the case of 110 ** latter crystallization temperature, the relative reflectance will be not less than 83%.
[0014]Record equivalent to at least 0.1 micrometer of record mark length is possible by extracting the beam system of recording laser light in this invention, since the recording material is a Sb3Te system recording material. As a recorded state stabilizing material in this invention, four group elements, 1B group element, and three group elements are effective. germanium is the most effective and also Cu, In, and B are effective. In addition, five fellows' N is also effective in recorded state stabilization. As a crystallization accelerating material, five group elements and six group elements are effective. In particular, Sb, Bi, and Te are effective.

[0015]A crystallization promoting layer contains one or more sorts of ******** of the above, a recorded state stabilizing material, a crystallization accelerating material, and high linear velocity-ized material. The layer which consists of 2 yuan, Bi and germanium, is the most preferred as a crystallization promoting layer. To a crystallization promoting layer, the further impurity element may be added for melting point adjustment etc. An usable material as an impurity element Ag, Cd, Ce, Co, Cr, Fe, H, Hg, Ir, K, La, Li, Mo, Na, nickel, O, P, Pb, Pd, Po, Pr, Pt, Pu, Rb, Rh, Ru, S, Se, Si, Sn, Sr, Th, Ti, Tl, U, Cl, Br, etc. are mentioned.

[0016]The above-mentioned crystallization promoting layer does not need to serve as a thin film [**** / completely] on a substrate. That is, in the case of about 1 nm, membrane formation thickness is the island shape of discontinuous a large number by mass membrane thickness. If membrane formation thickness increases, said islands will be connected and it will become a perfect thin film on a substrate. In this invention, said island shape is also called crystallization promoting layer in a microscopic meaning.

[0017]Next, an example of the optical information recording medium by this invention is shown in drawing 1. (1) is a substrate and (2) is the 1st dielectric layer and an organic protective layer by which, as for a reflection radiation layer and (7), the 2nd dielectric layer and (6) are provided [(3)] for a recording layer and (5) on a reflection radiation layer if needed in a crystallization promoting layer and (4). By a diagram, only the information board side of an optical information recording medium is shown. As for the material of a substrate, in almost all cases, polycarbonate is used. After substrate thickness forms the layer of drawing 1 in the case more than 1-mm thickness, and about 0.6-mm substrate, it is and, in more than 2.0-mm thickness, in the case of **0.3 mm and 1.0-mm thickness, the permissible error of the substrate thickness in this invention comes out of the case where paste other 0.6-mm boards together and it is referred to as about 1.2 mm, **0.2 mm in the case of **0.2 mm and 0.6-mm thickness. The slot is formed in the substrate and the depth is 200A - about 450A. The pitch of a slot is 0.74 micrometer when using it as DVD compatible media. If minuteness making of this pitch is carried out to about 0.3 micrometer, record of not less than 20 GB is possible on a disk 120 mm in radius by using the laser of blue light.

[0018]In this invention, as the 1st and 2nd dielectric layers, SiOx, ZnO, SnO_2, aluminum$_2O_3$, TiO_2, Metallic oxides, such as In_2O_3, MgO, ZrO_2, and Ta_2O_5, Carbide, such as sulfides, such as nitrides, such as Si_3N_4, AlN, TiN, BN, and ZrN, ZnS, and TaS_4, SiC, TaC, B_4C, WC, TiC, and ZrC, is mentioned. Such materials can be alone used as a protective layer, and can also be used as a mixture. For example, ZnS, SiOx, and Ta_2O_5 and SiOx are mentioned as a mixture.

[0019]The thickness of the 1st dielectric layer has the preferred range of 50-250 nm. If it becomes thinner than 50 nm, it becomes the fall of an environment-resistant protection feature, a heat-resistant fall, and the fall of ********, and is not desirable. in the film production process by a sputtering technique etc. if it becomes thicker than 250 nm -- film temperature -- since film peeling and a crack arise by the rise of a degree or the fall of the sensitivity at the time of record is brought about, it is not desirable. As for the thickness of the 2nd dielectric layer, 15-50 nm is preferred. In the case of the 2nd dielectric layer, if thinner than 10 nm, heat resistance falls and it is not desirable. On the contrary, if 100 nm is exceeded, repetitive overwriting characteristics will worsen due to the fall of the film peeling by the fall of recording sensitivity, and a rise in heat, modification, and heat dissipation nature.

[0020]As a reflection radiation layer (6), the simple substance or alloy of material mainly concerned with metal, such as aluminum, Au, Cu, Ag, Cr, Sr, Zn, In, Pd, Zr, Fe, Co, nickel, Si, germanium, Sb, Ta, W, Ti, and Pb, can be used. It is important for this layer to make heat radiate efficiently, and 50-160 nm of thickness is preferred. When thickness is too thick, radiation efficiency is too large, if sensitivity worsens and is too thin, sensitivity is good, but repetitive overwriting characteristics worsen. As the characteristic, thermal conductivity is high and it is required with a high-melting point that adhesion with the charge of a protective layer material should be good etc.

[0021]One in the case of pasting together to drawing 2 and using a substrate about 0.6 mm thick is shown. About dielectric materials, a crystallization accelerating material, a recording material, and the charge of a reflective radiating material, it is the same as that of the case of said drawing 1. In the case of drawing 2, transmissivity of the translucent reflection radiation layer of (13) and the 1st recording layer of (11) is made not less than 50%.

[0022]The case where it has a two-layer recording layer on a substrate about 1 mm thick is shown in drawing 3. About dielectric materials, a crystallization accelerating material, a recording material, and the charge of a reflective radiating material, it is the same as that of the case of said drawing 1. Since the transmissivity of the 1st recording layer is adjusted in the case of drawing 3, the 1st dielectric layer of (22) and the 2nd dielectric layer of (25) may be made into the multilayered constitution of a dissimilar material.

[0023]In order to carry out optical recording to the optical information recording medium of such this invention, when forming AMORUFASU equivalent to a reference clock length n cycle, laser pulse irradiation of a time (n-1) is performed at the time of n cycle. For example, when forming AMORUFASU of the length of reference clock 5 cycle shown in drawing 4, it irradiates with 4 times of laser pulses. in recording-linear-velocity 8.5 m/s -- the case (DVD.) of record with 63.7 MHz of reference clocks ** laser radiation start time delay in 2.4X record, for example, a figure, -- 19 ns and ** leading pulse width -- for 6 ns, for 7 ns, 15 mW and ** erase power are performed at 8 mW, and, as for ** multi-pulse width, ** cooling pulse width performs ** cooling power for ** record power at 0.1 mW for 14.5 ns.

[0024]For example, the above-mentioned material and the optical information recording medium by composition are the semiconductor lasers whose wavelength is 635 or 650 nm, and record reproduction can be carried out using the pickup of NA0.6. As a record method, a modulation code can use EFM or an EFM+ [8 / 16RLL (2, 10)] method by Pulse Width Modulation, for example. In this case, a pulse is divided into the multi-pulse part of a leading pulse and after that. A multi-pulse part is for repeating heating and cooling and performing them. In this case, the relation of each power serves as heating (record) power > erase power > cooling power, and cooling power is lowered to a read-out power grade. Usually, linear velocity is performed by 3.5 - 8.5 m/s, and read-out power is performed at 1 mW or less.

[Translation done.]

D4

(19)日本国特許庁（ＪＰ）　(12) 公開特許公報（Ａ）　(11)特許出願公開番号
特開2003－288737
（P2003－288737A）
(43)公開日 平成15年10月10日(2003.10.10)

(51)Int.Cl.7	識別記号	ＦＩ		テーマコード*(参考)
Ｇ１１Ｂ　7/24	５２２	Ｇ１１Ｂ　7/24	５２２Ｄ	２Ｈ１１１
	５１１		５１１	５Ｄ０２９
Ｂ４１Ｍ　5/26		Ｂ４１Ｍ　5/26		Ｘ

審査請求 未請求 請求項の数９　ＯＬ（全 ７ 頁）

(21)出願番号　特願2002－130158(P2002－130158)
(22)出願日　平成14年５月１日(2002.5.1)
(31)優先権主張番号　特願2002－17389(P2002－17389)
(32)優先日　平成14年１月25日(2002.1.25)
(33)優先権主張国　日本（ＪＰ）

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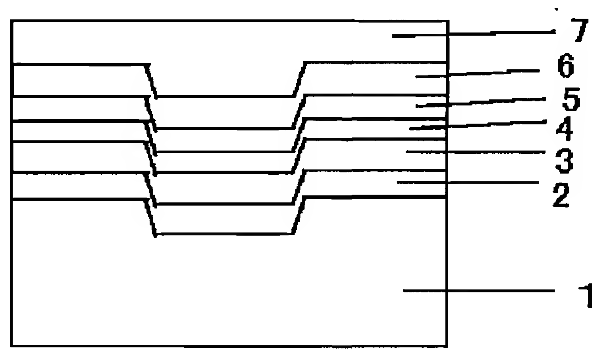
最終頁に続く

(54)【発明の名称】 光情報記録媒体

(57)【要約】
【課題】 高速オーバーライト記録に対応できる、ＤＶＤ－ＲＯＭ互換が可能な４．７ＧＢ／直径１２０mmディスク以上の記録密度の初期化レスメディアを実現すること。
【解決手段】 Ｓｂ、Ｔｅの他に、他元素を実質的に含まない又は周期律表第Ｉ族乃至第VII族に属する元素の少なくとも１種類の元素を含む記録層と、その余の層とを有する光記録媒体において、前記その余の層が高線速化材料を含み、前記光情報媒体にエネルギー照射して記録操作することにより、該記録層の製膜工程終了時に該記録層中に存在した以上の量の前記他元素が前記その余の層から記録層中に移行して存在するようになる相変化記録媒体。



【特許請求の範囲】

【請求項１】　Ｓｂ、Ｔｅの他に、他元素を実質的に含まない又は周期律表第Ｉ族乃至第VII族に属する元素の少なくとも１種類の元素を含む記録層と、その余の層とを有する光記録媒体において、前記その余の層が高線速化材料を含み、前記光情報媒体にエネルギー照射して記録操作することにより、該記録層の製膜工程終了時に該記録層中に存在した以上の量の前記他元素が前記その余の層から記録層中に移行して存在するようになる相変化記録媒体。

【請求項２】　前記記録層が、Ｓｂと、原子数でＳｂの１／３以下のＴｅとを含み、且つ０乃至５原子％未満のＧｅ含有量であることを特徴とする請求項１に記載の相変化記録媒体。

【請求項３】　前記その余の層が、記録状態安定化材料と結晶化促進材料とを含む結晶化促進層であることを特徴とする請求項１又は２に記載の相変化記録媒体。

【請求項４】　前記記録状態安定化材料が、４族元素、１Ｂ族元素、３族元素及び／又は５族元素であることを特徴とする請求項３に記載の相変化記録媒体。

【請求項５】　前記記録状態安定化材料が、Ｇｅ、Ｃｕ、Ｉｎ、Ｂ及び／又はＮであることを特徴とする請求項４に記載の相変化記録媒体。

【請求項６】　前記高線速化材料がＧａ、Ｄｙ、Ｃａ、Ｍｇ、Ｍｎからなる群から選ばれた少なくとも一つの元素であることを特徴とする請求項３に記載の相変化記録媒体。

【請求項７】　前記高線速化材料がＧａまたは／及びＭｎであることを特徴とする請求項６に記載の相変化記録媒体。

【請求項８】　前記結晶化促進材料が、５族元素、６族元素であることを特徴とする請求項３に記載の相変化記録媒体。

【請求項９】　前記結晶化促進材料が、Ｓｂ、Ｂｉ及び／又はＴｅであることを特徴とする請求項８に記載の相変化記録媒体。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、初期化操作が不要で、保存信頼性に優れ、高速オーバーライト記録に対応できる光情報記録媒体に関するものであり、光ディスクに応用される。

【０００２】

【従来の技術】レーザビーム照射により情報の記録、再生及び消去が可能な光情報記録媒体とレーザビーム照射により情報の記録、再生及び消去が可能な光情報記録媒体として、結晶状態と非晶質（アモルファス）状態の可逆的相変化を利用した相変化ＣＤ－ＲＷ、ＤＶＤ－ＲＡＭ、－ＲＷ、＋ＲＷメディアなどとしての商品化が期待されている。

【０００３】本発明者等は、上記相変化型光情報記録媒体のうち、記録層を空間群Ｆｍ３ｍに属する準安定Ｓｂ３Ｔｅ相を有することを特徴とする記録層で構成したものについて研究、開発を進めてきた（特開平１０－２１７０６９号公報等記載）。この準安定相はＳｂ－Ｔｅ共晶構造の記録層とは異なっており、ＳｂとＳｂ２Ｔｅ３とに分離せず、結晶粒界に起因する記録マークの乱れも生じない。そのため、空間群Ｆｍ３ｍに属する準安定Ｓｂ３Ｔｅ相を有することを特徴とする記録層を用いたものは、高密度記録が可能となる長所を持っている。さらに、空間群Ｆｍ３ｍに属する準安定Ｓｂ３Ｔｅ相を有することを特徴とする記録層は繰り返し記録時の熱衝撃に強いため、優れた繰り返し記録特性を有している。

【０００４】ところで、この空間群Ｆｍ３ｍに属する準安定Ｓｂ３Ｔｅ相を有することを特徴とする記録層を用いた相変化型光情報記録媒体では、記録層はスパッタリング、蒸着などの真空製膜法で製膜され、製膜直後の膜は、通常、非晶質状態となっている。一方、製品化された光情報記録媒体の記録層は結晶状態でなければならない。なぜなら、相転移を利用する書き換え型の光情報記録担体では、一般に記録膜が記録状態では非晶質状態に、消去（初期化）状態では結晶状態に設定されるからである。このため、記録層の製膜直後に、レーザビーム照射などの熱処理を施して記録層を結晶化させる初期化プロセスが必要であった。しかしながら、上記初期化プロセスには３０秒以上の時間が必要であるため、スループットがダウンし、量産を行なう場合には初期化プロセスのための装置が多数台必要となり、設備費が高価になる。その結果、製品コストの上昇を招くことになる。

【０００５】こうした問題に対して、Ｓｂ、Ｔｅの他に、他元素を実質的に含まない又は周期律表第Ｉ族乃至VII族に属する元素の少なくとも１種類の元素を含む記録層と、結晶化促進材料と記録状態安定化材料からなる結晶化促進層からなる、その余の層とを有する光記録媒体において、前記光記録媒体にエネルギー照射して記録操作することにより、該記録層の成膜工程終了時に該記録層中に存在した以上の量の前記他元素が前記その余の層から記録層中に移行して存在するようにして、初期化工程を不要とした技術がある（特願２００１－３１９８８７号明細書）。しかしながら、近年、相変化記録媒体は高密度画像記録への用途が拡大するとともに、より一層の高速オーバーライト実現（ＤＶＤ－ＲＯＭの再生線速の２～５倍速である７～１７ｍ／ｓ）が要求されるようになってきた。ここで、高速オーバーライト実現には、マーク消去時、すなわち融点近傍における記録層材料の結晶化速度の向上（以下、高速化）が必要となるが、前記技術では、そのような高速オーバーライトに対応することが困難である。なぜならば、これまで知られている知見によると、記録層が空間群Ｆｍ３ｍに属する準安定Ｓｂ３Ｔｅ相を有する相変化型光情報記録媒体

（特開２０００－４３４１５号公報）については、Ｓｂの配合比を高くすることにより記録層材料の高速化は図れるが、それにともない媒体の保存信頼性が低下してしまうため、実用的なレベルで、高速オーバーライト可能な媒体を提供することができないからである。

【０００６】

【発明が解決しようとする課題】従って、本発明の目的は、上記問題点を解消し、高速オーバーライト記録に対応できるＤＶＤ－ＲＯＭ互換が可能な４．７ＧＢ／直径１２０mmディスク以上の記録密度の初期化レスメディアを実現することにある。

【０００７】

【課題を解決するための手段】上記課題は、本発明の（１）「Ｓｂ、Ｔｅの他に、他元素を実質的に含まない又は周期律表第Ｉ族乃至第VII族に属する元素の少なくとも１種類の元素を含む記録層と、その余の層とを有する光記録媒体において、前記その余の層が高線速化材料を含み、前記光情報媒体にエネルギー照射して記録操作することにより、該記録層の製膜工程終了時に該記録層中に存在した以上の量の前記他元素が前記その余の層から記録層中に移行して存在するようになる相変化記録媒体」、（２）「前記記録層が、Ｓｂと、原子数でＳｂの１／３以下のＴｅとを含み、且つ０乃至５原子％未満のＧｅ含有量であることを特徴とする前記第（１）項に記載の相変化記録媒体」、（３）「前記その余の層が、記録状態安定化材料と結晶化促進材料とを含む結晶化促進層であることを特徴とする前記第（１）項又は第（２）項に記載の相変化記録媒体」、（４）「前記記録状態安定化材料が、４族元素、１Ｂ族元素、３族元素及び／又は５族元素であることを特徴とする前記第（３）項に記載の相変化記録媒体」、（５）「前記記録状態安定化材料が、Ｇｅ、Ｃｕ、Ｉｎ、Ｂ及び／又はＮであることを特徴とする前記第（４）項に記載の相変化記録媒体」、（６）「前記高線速化材料がＧａ、Ｄｙ、Ｃａ、Ｍｇ、Ｍｎからなる群から選ばれた少なくとも一つの元素であることを特徴とする前記第（３）項に記載の相変化記録媒体」、（７）「前記高線速化材料がＧａまたは／及びＭｎであることを特徴とする前記第（６）項に記載の相変化記録媒体」、（８）「前記結晶化促進材料が、５族元素、６族元素であることを特徴とする前記第（３）項に記載の相変化記録媒体」、（９）「前記結晶化促進材料が、Ｓｂ、Ｂｉ及び／又はＴｅであることを特徴とする前記第（８）項に記載の相変化記録媒体」により達成される。

【０００８】Ｓｂ、Ｔｅの他に、他元素を実質的に含まない又は周期律表第Ｉ族乃至第VII族に属する元素の少なくとも１種類の元素を含む記録層と、その余の層とを有する光記録媒体において、前記光記録媒体にエネルギー照射して記録操作することにより、該記録層の成膜工程終了時に該記録層中に存在した以上の量の前記他元素が前記その余の層から記録層中に移行して存在するようにし、その余の層を記録状態安定化材料と結晶化促進材料と高線速化材料とを含む結晶化促進層としたことにより、上記課題を解決したものである。

【０００９】

【発明の実施の形態】以下、本発明を詳細に説明する。本発明の特徴は、記録することにより、成膜工程終了時に記録層に存在した以上の量の副成分（以下、これを「不純物」とも云う）が記録層に存在するようになる相変化記録媒体であることである。本発明における主成分としてのＳｂ、Ｔｅとは、該副成分と対照的な量であり、記録層中のＳｂ及びＴｅがＳｂ３Ｔｅ系組成を維持し且つ記録層の記録材料が書込操作及び消去操作によりアモルファス相ー結晶相に相変換可能な程度の量比を保持することを意味する。結晶化促進層に不純物として記録状態安定化材料を含み、相変化記録時に前記不純物が記録層へと拡散し成膜工程終了時に記録層に存在した以上の量の不純物が記録層に存在するようになる。そのため、あらかじめ記録材料が含むべき記録状態安定化材料を減少させることができる。

【００１０】さらに、本発明では、結晶化促進層に不純物として高線速化材料を含む。高線速化材料は、融点近傍における記録材料の結晶化速度を高める効果を有するものであり、高速オーバーライト実現（ＤＶＤ－ＲＯＭの再生線速の２～５倍速である７～１７m／ｓ）が可能となる。なお、高線速化材料を加えることにより、記録層に占めるＳｂの割合を増やすことなく、記録層材料の高速化を図れるため、記録層材料の高速化にともなって保存信頼性が低下してしまうという問題が生じない。

【００１１】高線速化材料としては、一群Ｇａ、Ｄｙ、Ｃａ、Ｍｇ、Ｍｎの中から選ばれる少なくとも一つの元素を用いることが好ましい。これらは、Ｓｂ３Ｔｅ相を有する相変化型光情報記録媒体に対する添加効果を周期律表上の数多くの元素について調べた結果から、記録層材料の高速化の効果が確認されたものである。

【００１２】なお、一群の中でもＧａおよび／またはＭｎが特に好ましい。Ｄｙ、Ｃａ、Ｍｇはいずれも高速化は図れるものの、多量に含有させた場合、繰り返し記録特性の低下をきたすという問題を有している。Ｇａは、Ｄｙ、Ｃａ、Ｍｇと同様に繰り返し記録特性の低下をきたすという問題を有しているものの、保存特性を向上させる効果を有している。このため、高速化とともに記録状態の安定化も図ることができる。一方，Ｍｎは、保存特性を向上させる効果はGaほどではないが、多量に含有させた場合でも繰り返し記録特性の低下をきたすことがない。これらのことから、高線速化材料としてＧａおよびまたはＭｎを用いることにより、繰り返し特性が良く、保存特性がさらに優れた相変化記録媒体を実現することが可能である。

【００１３】結晶化促進層に含む記録状態安定化材料と

してＧｅを用いる場合、記録材料に含有させるＧｅは５原子％未満で良い。Ｓｂ３Ｔｅ系記録層において結晶記録層において記録材料に含有させるＧｅが５原子％未満の場合、結晶化温度が１６０℃未満となる。結晶化温度が１６０℃未満の場合は、結晶化促進層を用いることによりポリカーボネート基板の塑性変形温度以下の成膜プロセスでも相対反射率８０％の結晶記録膜の形成が可能となる。結晶記録層形成のためのより望ましくはＧｅが３原子％未満であり、最も望ましくはＧｅを含有しない記録材料である。ここで、記録材料中に含まれるＧｅが１原子％未満の場合をＧｅを実質的に含有しないと定義する。Ｓｂ３Ｔｅ系記録材料に含まれるＧｅが３原子％の場合、結晶化温度は１４７℃、１原子％未満の場合は１１０℃である。前者結晶化温度１４７℃の場合、前述のポリカーボネート基板耐熱温度の６７℃でも初期化レスで使用可能な結晶記録膜となる。その相対反射率は８０％程度である。後者結晶化温度１１０℃の場合は、基板温度６７℃でも更に完全な結晶膜となり、その相対反射率は８３％以上となる。

【００１４】本発明では、記録材料がＳｂ３Ｔｅ系記録材料であることから、記録レーザー光のビーム系を絞ることにより少なくとも０．１μｍの記録マーク長に相当する記録までは可能である。本発明における記録状態安定化材料としては、４族元素、１Ｂ族元素、３族元素が有効である。Ｇｅが最も有効である他、Ｃｕ、Ｉｎ、Ｂも有効である。加えて、５族のＮも記録状態安定化に有効である。結晶化促進材料としては、５族元素、６族元素が有効である。特に、Ｓｂ、Ｂｉ、Ｔｅが有効である。

【００１５】結晶化促進層は、上記、記録状態安定化材料と結晶化促進材料と高線速化材料とのぞれぞれ１種以上を含有する。結晶化促進層として最も好ましいのは、ＢｉとＧｅの２元からなる層である。更に、結晶化促進層には、融点調整等のために更なる不純物元素を添加する場合もある。不純物元素として使用可能な材料は、Ａｇ、Ｃｄ、Ｃｅ、Ｃｏ、Ｃｒ、Ｆｅ、Ｈ、Ｈｇ、Ｉｒ、Ｋ、Ｌａ、Ｌｉ、Ｍｏ、Ｎａ、Ｎｉ、Ｏ、Ｐ、Ｐｂ、Ｐｄ、Ｐｏ、Ｐｒ、Ｐｔ、Ｐｕ、Ｒｂ、Ｒｈ、Ｒｕ、Ｓ、Ｓｅ、Ｓｉ、Ｓｎ、Ｓｒ、Ｔｈ、Ｔｉ、Ｔｌ、Ｕ、ＣｌおよびＢｒ等が挙げられる。

【００１６】上記結晶化促進層は基板上で完全に連続な薄膜とならなくても良い。すなわち、成膜膜厚が質量膜厚で１ｎｍ程度の場合は、不連続な多数の島状になっている。成膜膜厚が増加すると、前記島同士がつながり、基板上で完全な薄膜となる。本発明においては、前記島状をも微視的な意味で結晶化促進層という。

【００１７】次に、本発明による光情報記録媒体の一例を図１に示す。（１）が基板、（２）が第１の誘電体層、（３）が結晶化促進層、（４）が記録層、（５）が第２の誘電体層、（６）が反射放熱層、（７）は必要に応じて反射放熱層の上に設けられる有機保護層である。なお、図では、光情報記録媒体の情報基板側のみを示している。基板の材料は、ほとんどの場合ポリカーボネートが使用される。基板厚さは、１mm厚以上の場合と、約０．６ｍｍの基板に図１の層を形成した後、他の０．６ｍｍ基板を貼り合わせて約１．２ｍｍとする場合があり、本発明における基板厚の許容誤差は、２．０mm厚以上の場合±０．３ｍｍ、１．０mm厚の場合±０．２ｍｍ、０．６ｍｍ厚の場合±０．２ｍｍ、である。基板には、溝が形成してあり、その深さは２００Å～４５０Å程度である。溝のピッチはＤＶＤ互換メディアとして使用する場合０．７４μｍである。このピッチを、０．３μｍ程度まで微細化すると、青色発光のレーザーを使うことにより、半径１２０ｍｍのディスク上で２０ＧＢ以上の記録が可能である。

【００１８】本発明において、第１および第２の誘電体層としては、ＳｉＯｘ、ＺｎＯ、SnO_2、Al_2O_3、TiO_2、In_2O_3、ＭｇＯ、ZrO_2、Ta_2O_5 等の金属酸化物、Si_3N_4、ＡｌＮ、ＴｉＮ、ＢＮ、ＺｒＮ等の窒化物、ＺｎＳ、TaS_4 等の硫化物、ＳｉＣ、ＴａＣ、B_4C、ＷＣ、ＴｉＣ、ＺｒＣ等の炭化物が挙げられる。これらの材料は、単体で保護層として用いることができ、また、混合物として用いることもできる。例えば、混合物としては、ＺｎＳとＳｉＯｘ、Ta_2O_5 とＳｉＯｘが挙げられる。

【００１９】第１の誘電体層の膜厚は、５０～２５０ｎｍの範囲が好ましい。５０ｎｍより薄くなると、耐環境性保護機能の低下、耐熱性低下、畜熱効果の低下となり好ましくない。２５０ｎｍより厚くなると、スパッタ法等による製膜過程において、膜温度の上昇により膜剥離やクラックが生じたり、記録時の感度の低下をもたらすので好ましくない。第２の誘電体層の膜厚は１５～５０ｎｍが好ましい。第２の誘電体層の場合、１０ｎｍより薄いと耐熱性が低下し好ましくない。逆に、１００ｎｍを越えると、記録感度の低下、温度上昇による膜剥離、変形、放熱性の低下により、繰り返しオーバーライト特性が悪くなる。

【００２０】反射放熱層（６）としては、Ａｌ、Ａｕ、Ｃｕ、Ａｇ、Ｃｒ、Ｓｒ、Ｚｎ、Ｉｎ、Ｐｄ、Ｚｒ、Ｆｅ、Ｃｏ、Ｎｉ、Ｓｉ、Ｇｅ、Ｓｂ、Ｔａ、Ｗ、Ｔｉ、Ｐｂ等の金属を主とした材料の単体または合金を用いることができる。この層は、熱を効率的に放散させることが重要であり、膜厚は５０～１６０ｎｍが好ましい。膜厚が厚すぎると、放熱効率が大きすぎて感度が悪くなり、薄すぎると感度は良好であるが、繰り返しオーバーライト特性が悪くなる。特性としては、熱伝導率が高く、高融点で保護層材料との密着性がよいことなどが要求される。

【００２１】図２に、厚さ約０．６ｍｍの基板を貼り合わせて使用する場合の１例を示す。誘電体材料、結晶化

促進材料、記録材料、反射放熱材料については、前記図１の場合と同様である。図２の場合では、（１３）の半透明反射放熱層及び、（１１）の第１の記録層の透過率を５０％以上にする。

【００２２】図３に、厚さ１mm程度の基板上に、２層の記録層を有する場合を示す。誘電体材料、結晶化促進材料、記録材料、反射放熱材料については、前記図１の場合と同様である。図３の場合、第１の記録層の透過率を調節するため、（２２）の第１の誘電体層及び（２５）の第２の誘電体層を異種材料の多層構成とする場合がある。

【００２３】このような本発明の光情報記録媒体に光記録するには、基準クロック長さn周期に相当するアモルファスを形成する場合、n周期の時間に（n－１）回のレーザーパルス照射を行なう。例えば、図４に示す基準クロック５周期相当の長さのアモルファスを形成する場合は、４回のレーザーパルスを照射する。記録線速度８．５m／sにおいて、基準クロック６３．７MHzでの記録の場合（DVD、２．４倍速記録）、例えば図における、①レーザー照射開始遅延時間は１９ns、②先頭パルス幅は６ns、③マルチパルス幅は７ns、④冷却パルス幅は１４．５ns、⑤記録パワーは１５mW、⑥消去パワーは８mW、⑦冷却パワーは０．１mWにて行なう。

【００２４】上記材料および構成による光情報記録媒体は、例えば、波長が６３５あるいは６５０nmの半導体レーザーで、NA０．６のピックアップを用い記録再生することができる。記録方法としては、例えば、Pulse Width Modulationで変調コードがEFM又はEFM＋［８／１６RLL（２，１０）］方式等を用いることができる。この場合、パルスは先頭パルスとその後のマルチパルス部に分かれる。マルチパルス部は、加熱、冷却を繰り返し行なうためのものである。この場合、各パワーの関係は、加熱（記録）パワー＞消去パワー＞冷却パワーとなっていて、冷却パワーは読み出しパワー程度まで下げる。通常、線速は３．５～８．５m／s、読み出しパワーは１mW以下で行なう。

【００２５】

【実施例】以下、本発明を実施例に基づき説明する。

０．６mm厚、直径５インチのポリカーボネート基板（以下、PC基板）上にスパッタリング法により以下の（１）～（５）の薄膜を形成する。

（１）第１の誘電体層：ZnS・SiO2（ターゲットのmol比で７９．５：２０．５）；膜厚２２０nm。

（２）結晶化促進層：記録状態安定化材料、結晶化促進材料、高線速化材料からなるターゲットを用いた。組成及び膜厚は表１に示す。

（３）記録層：Sb3Te系材料の合金ターゲット用いた。組成は表１に示す。膜厚１６nm。

（４）第２の誘電体層：ZnS・SiO_2（ターゲットのmol％比で７９．５：２０．５）；膜厚１０nm。

（５）反射放熱層：AlTi；膜厚１４０nm。

記録層成膜時の基板温度は、ポリカーボネート基板が塑性変形しない限界の温度で行なった。この場合、基板に接触させた熱電対が１００℃となるようにIRランプ出力を制御して、熱電体によるモニター温度が安定した後に基板を記録層成膜室に搬送している。記録層成膜室での、基板温度を測定すると６７℃であった。成膜終了後、UV硬化樹脂をスピンコートした後、UV光照射により硬化形成させた。このようにして製造した基板を、他の厚さ０．６mm基板と貼り合わせて約１．２mm厚とした。

【００２６】波長６６０nm、NA０．６５のピックアップヘッドを使用して、記録線速度８．５、１４m／sの速さで、直径１２０mmディスク上で４．７GBとなる記録密度相当の記録を行なった。１つのマークを書く際にも、レーザー照射と冷却を繰り返すマルチパルス方式で記録を行なった。クロック１周期の倍数の長さのアモルファスマークを形成するのであるが、マーク長あたりのクロックの振動数と同一の回数のレーザー照射と冷却を行なった。アモルファスマーク形成時のレーザー照射パワー（記録パワー）と、結晶スペース形成時のパワー（消去パワー）の比は、結晶化促進層の膜厚及び材料により最適値が異なり、（記録パワー）／（消去パワー）＝０．４～０．６である。

【００２７】記録特性中、ジッターは、記録マークとスペースの境界の読み出し時間のズレの標準偏差を、読み出しクロック１周期時間で除した値であり（単位％）、モジュレーションは、１４Tの振幅を１４Tの反射率で除した値である。

【００２８】（実施例１～８、比較例１～２）実施例１～８、比較例１～２について、製造直後のメディア反射率および相対反射率、記録線速度８．５、１４m／sの速さ（線速DVD２．５、４倍速相当）で評価した繰り返し記録ジッタ特性、８０℃保管時に記録ジッターが１％上昇する時間を評価した。その結果を表２に示す。なお、ここで相対反射率とは、製膜段階での結晶化進行度の目安となるものであり、以下の式で定義される。

【００２９】

【数１】相対反射率＝(記録前のメディア反射率)／(記録時に形成される溶融再結晶の反射率)×１００（％）

【００３０】そして発明者が得た知見によると、これが８０％未満であると、メディア反射率が記録とともに変動するため、良好なメディア特性が得られない。

【００３１】評価した結果、実施例１～８については、メディア反射率は２０％以上、相対反射率は８０％以上を確保でき、また保存信頼性としては１００時間の耐久時間を確保できており、保存信頼性が良好であった。またさらに、記録線速度８．５、１４m／sの速さでの繰り返し記録ジッタ特性は、初回、１０００回記録ともに

１０％未満であり、良好な特性を示した。

【００３２】これに対して、比較例１においては、保存信頼性が良好であり、記録線速度８．５ｍ／ｓでの繰り返し記録ジッタ特性は、初回、１０００回記録ともに１０％未満であり、良好な特性を示したが、１４ｍ／ｓの記録線速度での繰り返し記録ジッタ特性は初回、１０００回記録ともに１５％を越えており、非常に悪い特性であった。これは、高線速化材料がないため、記録材料の高速化が不充分なためと思われる。　＊

＊【００３３】また、比較例２においては、記録線速度８．５、１４ｍ／ｓの速さでの繰り返し記録ジッタ特性は、初回、１０００回記録ともに１０％未満であり、良好な特性を示したものの、保存信頼性が非常に悪かった。これは、記録層材料の高速化のためＳｂの割合を増やしたために保存信頼性が低下したためと思われる。

【００３４】

【表１】

	結晶化促進層材料	結晶化促進層膜厚/nm	記録層材料	混合後組成 / atom%				
				Bi	Ge	X	Sb	Te
比較例1	BiGe	2.0	Sb80Te20	4.4	6.6	0	71.2	17.8
比較例2	BiGe	2.0	Sb86Te14	4.4	6.6	0	76.5	12.5
実施例1	BiGeGa	2.0	Sb80Te20	3.7	3.7	Ga:3.7	71.2	17.8
実施例2	BiGeGa	2.0	Sb80Te20	4.4	3.3	Ga:3.3	71.2	17.8
実施例3	BiGeDy	2.0	Sb80Te20	3.7	3.7	Dy:3.7	71.2	17.8
実施例4	BiGeMg	2.0	Sb80Te20	3.7	3.7	Mg:3.7	71.2	17.8
実施例5	BiGeCa	2.0	Sb80Te20	3.7	3.7	Ca:3.7	71.2	17.8
実施例6	BiGeGa	1.5	Ge2Sb78Te20	4	4.3	Ga:2.2	71.2	18.3
実施例7	BiGeMn	2.0	Sb80Te20	3.7	3.7	Mn:3.7	71.2	17.8
実施例8	BiGeGaMn	2.0	Sb80Te20	3.3	3.3	Ga:2.2 Mn2.2	71.2	17.8

【００３５】　※

※【表２】

	製造直後のメディア反射率／相対反射率	80℃保管時に記録ジッターが1%上昇する時間	初回記録／ダイレクトオーバーライト1000回目のジッター（記録線速 8.5m/s）	初回記録／ダイレクトオーバーライト1000回目のジッター（記録線速 14m/s）
比較例1	20%／83%	100時間以上	10%未満／10%未満	15%以上／15%以上
比較例2	20%／82%	5時間未満	10%未満／10%未満	10%未満／10%未満
実施例1	20%／81%	100時間以上	10%未満／10%未満	10%未満／10%未満
実施例2	20%／85%	100時間以上	10%未満／10%未満	10%未満／10%未満
実施例3	20%／83%	100時間以上	10%未満／10%未満	10%未満／10%未満
実施例4	20%／83%	100時間以上	10%未満／10%未満	10%未満／10%未満
実施例5	20%／83%	100時間以上	10%未満／10%未満	10%未満／10%未満
実施例6	20%／81%	100時間以上	10%未満／10%未満	10%未満／10%未満
実施例7	20%／83%	100時間以上	10%未満／10%未満	10%未満／10%未満
実施例8	20%／81%	100時間以上	10%未満／10%未満	10%未満／10%未満

【００３６】

【発明の効果】以上、詳細かつ具体的な説明から明らかなように、本発明により、Sb_3Te系記録材料相変化記録メディアの初期化レス製造を可能として、ＤＶＤ－ＲＯＭ互換が可能な４．７ＧＢ／直径１２０ｍｍディスク以上の記録密度の初期化レスメディアを実現できるという極めて優れた効果を奏するものである。

【図面の簡単な説明】

【図１】本発明による光情報記録媒体の一例を示した図である。

【図２】本発明による光情報記録媒体の一例を示した別の図である。

【図３】本発明による光情報記録媒体の一例を示した更に別の図である。

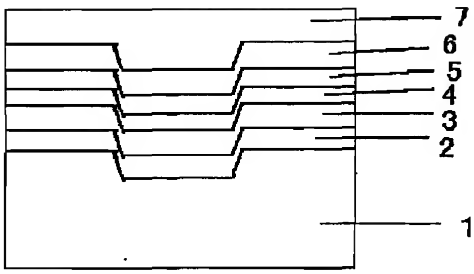
【図４】本発明の光情報記録媒体への記録モードの１例を示した図である。

【符号の説明】

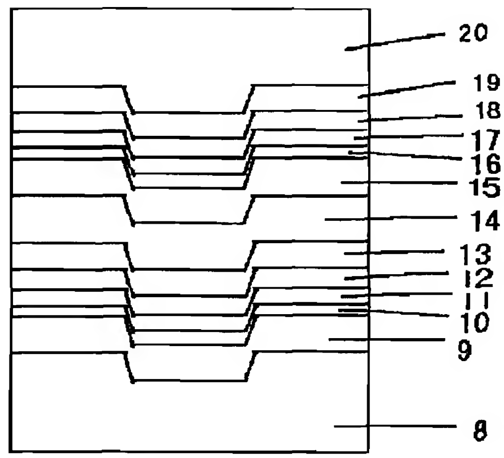
１　基板
２　第１の誘電体層
３　結晶化促進層
４　記録層
５　第２の誘電体層
６　反射放熱層
７　保護層
８　基板
９　第１の誘電体層
１０　結晶化促進層
１１　第１の記録層
１２　第２の誘電体層
１３　半透明反射放熱層
１４　保護層
１５　第３の誘電体層
１６　第２の記録層
１７　結晶化促進層
１８　第２の誘電体層
１９　反射放熱層

２０　基板
２１　カバー層
２２　第１の誘電体層
２３　結晶化促進層
２４　第１の記録層
２５　第２の誘電体層
２６　保護層
＊２７　第３の誘電体層
２８　第２の記録層
２９　結晶化促進層
３０　第４の誘電体層
３１　反射放熱層
３２　基板
＊

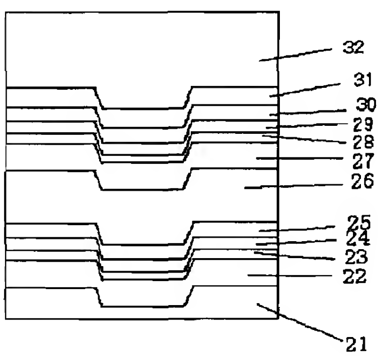
【図１】



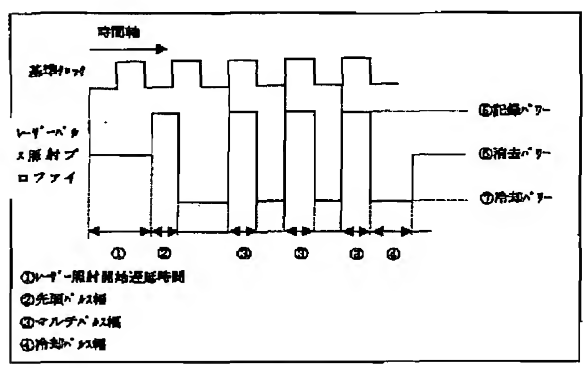
【図２】



【図３】



【図４】



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Ｆターム(参考)　2H111 EA04 EA12 EA23 EA32 EA33 FA01 FA11 FA23 FB05 FB09 FB12 FB30
5D029 JA01 JB03 JB05 JC09